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Effect of continuous cooling heat treatment on interface characteristics of S45C/copper compound casting

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The bonding of an S45C steel insert to copper during cast welding and continuous cooling treatment (including: furnace cooling; air cooling; oil quenching and water quenching) was investigated. The interface shear strength was determined using a push-out test. A cast welding layer formed between S45C steel and copper. After continuous cooling heat treatment, a cast welding layer was present near the S45C steel, an irregular layer near the copper matrix, and a middle layer between these two. X-ray diffraction analysis was used to determine that the interface layer consisted of carbon and $CuFeO_2$.Electron probe microanalysis (EPMA) demonstrated that most iron atoms and carbons diffused into the copper matrix. The interface shear strength of the compound casting under furnace-cooling was the largest and that of the compound casting formed by water-quenching was the smallest, and all fractures in the cast welding layer occurred near the S45C steel matrix.

1. Introduction

The wettability between two kinds of metals or a metal and a nonmetal is important in determining bonding between the two materials [1]. The characteristics of the materials, such as diffusion temperature and diffusion time are also important [2]. According to Durrant *et al*. [3], when mild steel by hot dipping and vacuum plasma spraying (VPS) to form separate Al-10% Fe and Ti-reacted layers, the wettability by an aluminum alloy was increased.

According to Shieu *et al*. [4], the formation of a solid solution interlayer at the interface between NiO and Pt increases the interface shear stress. According to Choi *et al*. [5], the reaction between Ti and SiC is thermodynamically driven. According to Lu *et al*. [6], the formation of a hard and brittle intermetallic compound and cavities in the interface, the fracture toughness would be reduced. According to the report of Evans *et al*. [7], on the cracking of interfaces, two constants dominate the appearance of cracks—the energy release rate and the mode mixity angle: (ϕ). (Definition: $\phi = 0$ implies open cracks; $\phi = \pi/2$ implies shear cracks).

Klomp [8] showed that heat treatment under different conditions yielded various bonding strengths in the interface of composites. Using austempering heat treatment, Moore *et al*. [9] changed the distribution and the size of ferrite iron grains and produced carbon steel, to forma material with the ductility of austenite steel, but with high strength and high toughness. According to Ma Qian *et al*. [10] who put steel wire into molten cast iron, some characteristics of austenite steel or of the phase transformation remained in the interface between the steel and iron phases.

Pushing-out and pulling-out tests are frequently used to determine interface shear strength [11–13]. Lewis *et al*. elucidated the microstructure of the interface using scanning electron microscopy and transmission electron microscopy; determined the interface strength in a pushing-out test, and then analyzed the composition of the interface using EDS, ELS and XPS [14]. According to Durrant [3], the shear stress at the interface is $L/[\pi D(t-d)]$, where L is the surface area of the inserted reinforcement; D is the diameter of the inserted reinforcement; t is the thickness of the specimen, and d is the measured insert displacement, excluding elastic distortion.

In this research, after pure copper and S45C hypereutectoid steel were cast-welded, four continuous cooling heat treatments, including quenching in water, quenching in oil, cooling in air and cooling in a furnace, were applied to the compound casting. Heat treatment was used to increase the hardness of the S45C steel matrix and the shear strength of the interface between S45C steel and pure copper. The microstructure of the compound casting was observed by OM (optical microscope) and SEM (scanning electron microscope) to determine the changes in the composition and the microstructure of the S45C steel and interface during heat

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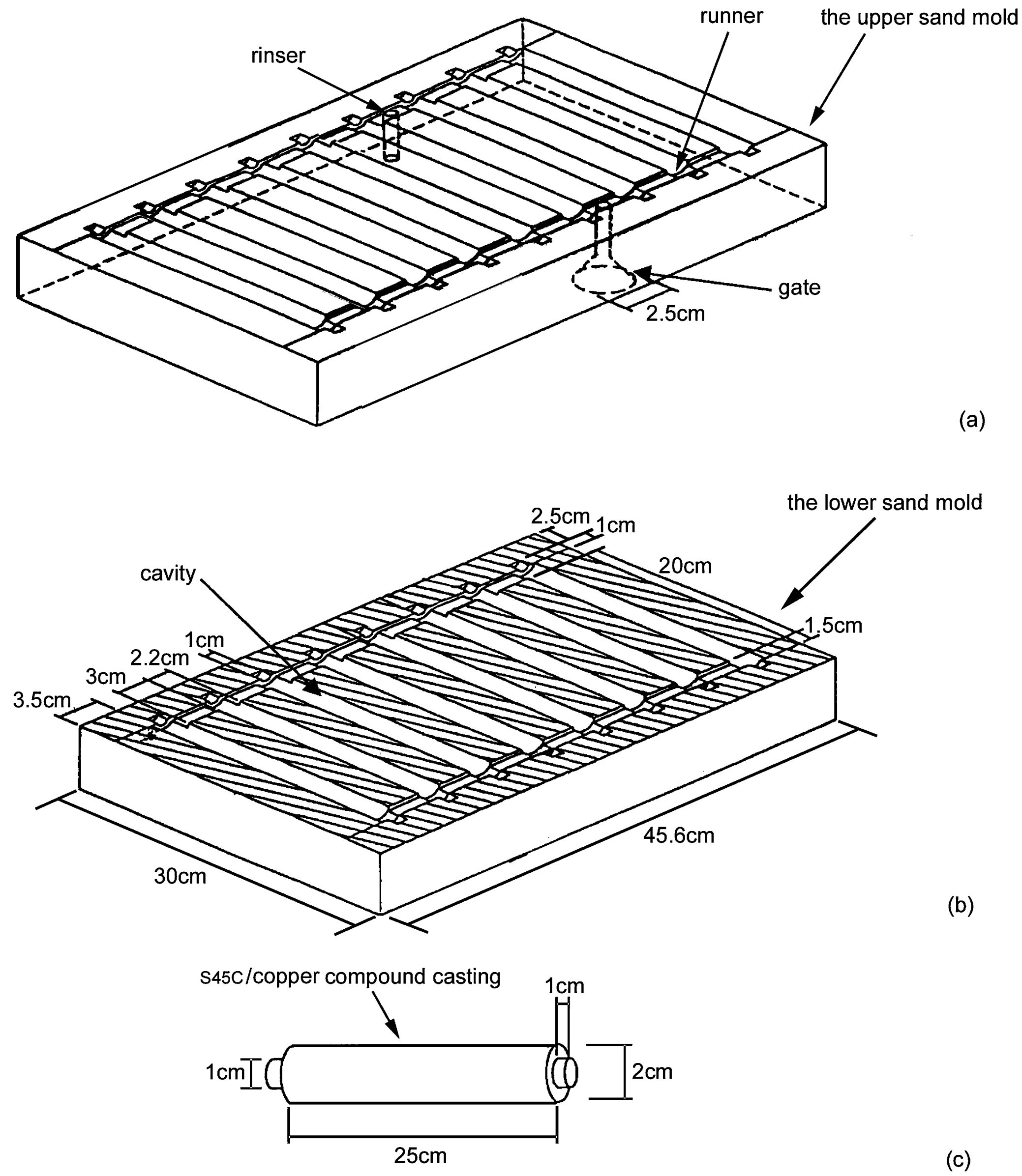

Figure 1 (a) to (c) Apparatus used in cast welding and compound casting.

treatment. The interface phase was analyzed by X-ray diffraction and the composition was determined using EDS and EPMA (electron probe micro-analysis). A push-out test was used to determine the interface shear strength.

2. Experimental procedure

2.1. Preparing materials

The matrix was 99.98% copper; S45C steel with a diameter of 10 mm and a length of 240 mm was the reinforcement to be cast-welded. Before it was cast-welded, the surface of S45C steel was polished using 1200 grit paper; oxides and grease on the surface S45C steel were eliminated by washing in alkaline solution (composing 16 wt% $NaCO_3$ with H_2O, and the balance is alkaline) at a washing temperature of 90°C (±5°C). The rod was cleaned in water; excess water on the surface of the steel rod was quickly eliminated using compressed argon gas. A compound casting was formed by cast welding the treated S45C steel to molten copper at 1150°C. Argon gas was used to prevent the surface oxidation of S45C steel. Fig. 1 shows the cast welding and compound casting apparatus.

2.2. Heat treatment

The compound casting was cut into specimens with a diameter of 20 mm and a thickness of 15 mm, preheated to 800°C (±3°C), and then isothermally heated for two hours to determine the effect of the heat treatment conditions on the interface. Subsequently, four continuous cooling heat treatments, including quenching in water [cooling by stirring in icy water for 60 s at 10°C (±2°C)], quenching in oil [cooling by stirring in an oil bath at 27°C ± 3°C for 90 s under isothermal conditions], cooling in air to 27°C ± 3°C and cooling in a furnace to 27°C ± 3°C were used. Table I shows the properties of the quenching oil.

2.3. Microstructural observations

A cut cross-section of a heat-treated compound casting was ground using 100 grit to 1200 grit carbimet papers,

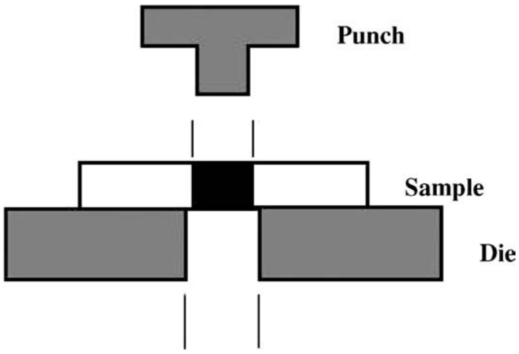

Figure 2 Schematic diagram of the push-out testing equipment.

polished using a suspension liquid of 0.3 μm Al_2O_3 particles, etched, and then using a solution of (95% ethanol +5% nitric acid) for three seconds. The microstructure of the S45C steel and the interface were observed using OPTIPHOT-100 Nikon OM (Nikon Corp., Tokyo) and Jeol-JSM 840A SEM (Japan Electron Optics Ltd., Tokyo). The relative proportion of iron to copper in the interface was analyzed by scanning lines using a Jeol JXA-8800M EPMA (Jeol USA, Inc., MA).

TABLE I Properties of the quenching oil

Quenching oil (TQ1) of Chinese Petroleum Corp.	
Gravity, D287 15.6°C API	30.5
Viscosity kin: cSt 40°C (100°C)	28.0 (5.10)
Viscosity index, D2270	106
Flash point, COC, °C (°F)	228 (442)
Fire point, COC, °C (°F)	240 (464)
Pour point, COC, °C (°F)	−7.7 (18)
Color D1500	2.5

Each interface formed during cast welding and heat-treating was removed and ground into a powder for

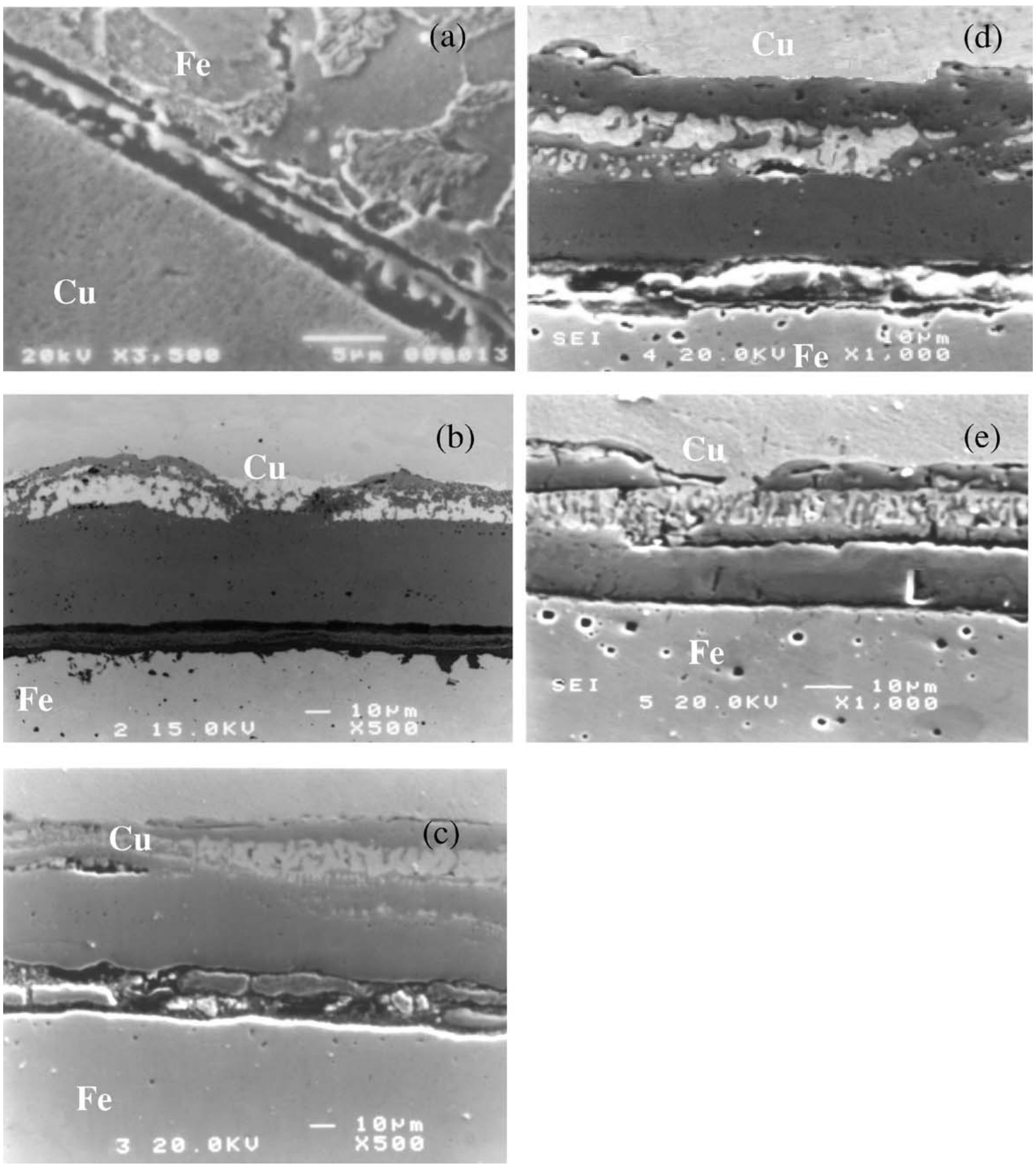

Figure 3 SEM photograph of S45C steel/copper compound casting: (a) before heat treatment, (b) after water quenching, (c) after oil quenching, (d) after air cooling, and (e) after furnace cooling.

X-ray diffraction analysis, using a MAC Science MXP-3TXT-A104 X-ray diffraction analyzer (Shimadzu Corp., Kyoto) with a 3 KW X-ray generator, a copper target, a wavelength of 1.54056A, a voltage of 40 KV and a current of 30 mA. The scanning from 20° to 80° was at 4°/min.

2.4. Push-out test

The testing rig for the push out tests consisted of an SKD11 (HRC62) tool and lower steel plate with a hold with a diameter of 10.2 mm hole, as shown in Fig. 2. A 250 mm thick disc obtained from the heat-treated compound casting was placed on the plate, and an SKD11 (HRC62) tool steel punch with a diameter of 9.8 mm was used to push out the S45C steel insert. The rig was used on an HT-9102 Instron testing machine (Hung Ta Instrument Co., Ltd., Taiwan). The S45C steel of the compound casting was pushed out at a crosshead speed of 5 mm/min.

2.5. Observing morphology fracture

The morphology of the fracture of the pushed-out specimen was observed using an OPTIPHOT-100 Nikon OM. The fracture morphology of the interface around the fractured zone, which could not be observed using OM, was observed after the specimen was polished and etched, but before testing.

3. Results and discussion

3.1. Microstructural observation

After cast welding, the microstructure of the S45C steel and the copper were as shown in Fig. 3a. An interface, called the "cast welding layer," formed between the S45C steel and copper because the molten copper at 1150°C formed a reactive layer while it was being poured into the pre-heated sand mold.

After S45C steel and copper were cast-welded together, and a continuous cooling heat treatment was applied to the interface, as shown in Fig. 3b to e, the interface was divided into three main layers. The layer closest to the S45C steel was the cast-welded layer (I); the one closest to the copper was an irregular layer (III); the other in between these two was the middle layer (II).

The water-quenched specimen contained a martensite phase, oil-quenching consisted of a mixture of fine pearlite and martensite; the air-cooled specimen contained medium pearlite, and the furnace-cooled specimen consisted of a mixture of coarse pearlite and spheroid cementite, as shown in Fig. 4.

3.2. X-ray diffraction and EPMA

A cast-welded layer produced by welding S45C steel to copper, was removed and ground into powder for X-ray diffraction analysis and the determination of the compositions of the chemical compounds such as $CuFeO_2$ and C, which yield the peak values shown in Fig. 5a. The interface layer, after continuous cooling heat treatment, was analyzed, the results were the same as those before treatment, as shown in Fig. 5b. Copper atoms and iron atoms have interdiffused to form $CuFeO_2$ and the decarbonization of the S45C steel surface is the source of carbon in the interface. Fig. 6a to d present the results of the EPMA qualitative analysis, the line-profile and the corresponding SEM iron and copper elemental mappings of cast-welded S45C steel and copper. These results imply that the iron content was more than the

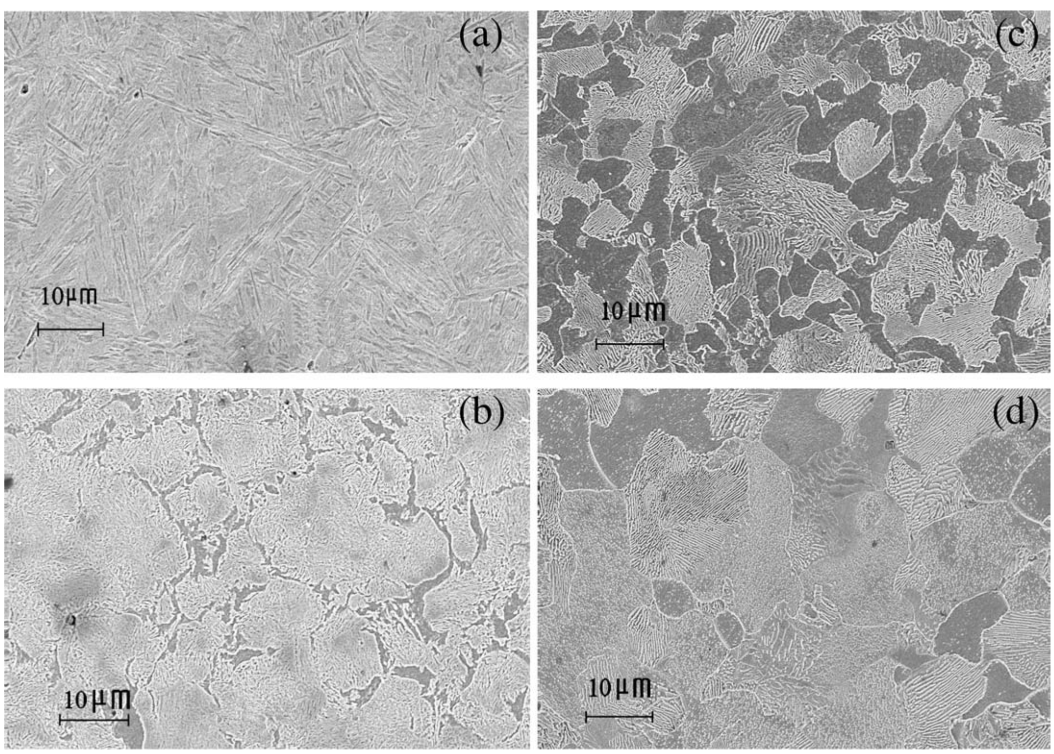

Figure 4 SEM photograph of S45C steel following: (a) water quenching, (b) oil quenching, (c) air cooling, and (d) furnace cooling.

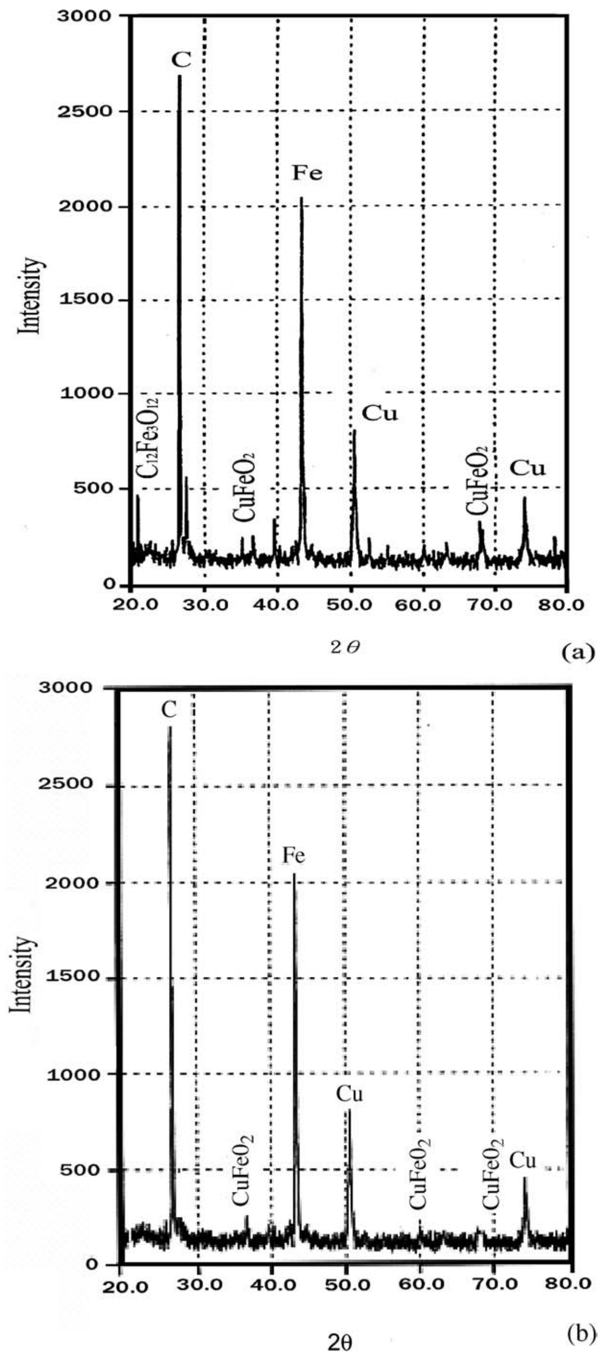

Figure 5 X-ray analysis of (a) S45C steel/copper compound casting and (b) after water quenching.

copper content in the cast-welding layer. After annealing for two hours at 760°C, and air cooling heat treatment, as shown in Fig. 7a to d, EPMA was applied after water quenching, oil quenching or furnace cooling. The results were the same as those obtained before, which the EPMA was applied after furnace cooling as presented in Fig. 8a to d. The results found that iron atoms tend to diffuse mainly into the copper matrix because the high concentration of carbon in the cast-welding layer effectively prevents the diffusion of copper atoms into the S45C steel matrix. Many iron atoms diffused into the copper matrix, so many holes were found in the S45C steel closest to the cast-welded layer. Water-quenched samples had the smallest holes and furnace-cooled samples included the largest holes, as shown in Fig. 9a and b, respectively.

TABLE II The contents of iron and copper in S45C steel and copper compound in the interface layer

S45C/copper		Fe (%)	Cu (%)	C (%)
Before continuous cooling treatment	Cast welding layer	64.31	1.08	34.61
Water quenching	Cast welding layer	57.15	0.19	42.66
	Middle layer	73.36	0.29	26.35
	Irregular layer	71.02	1.62	27.36
Oil quenching	Cast welding layer	43.96	0.65	55.39
	Middle layer	75.29	0.26	24.45
	Irregular layer	71.02	1.62	27.36
Air cooling	Cast welding layer	43.85	0.83	55.32
	Irregular layer	70.78	1.23	27.99
Furnace cooling	Cast welding layer	46.63	1.08	52.29
	Middle layer	72.38	0.11	27.51
	Irregular layer	70.20	1.55	28.25

Table II lists the contents of iron and copper in S45C steel and copper compounds in the continuously cooled heat-treated cast welding layer, the middle layer and the irregular layer. EPMA revealed that throughout the process of cast welding and continuous cooling heat treatment, the diffusion of iron atoms into the copper matrix dominated, further producing copper-iron compounds. However, very little copper diffused into the steel matrix. The cast-welded layer contained the most carbon. The middle layer had a similar content of carbon as an irregular layer. That is, the cast-welded layer in the solid solution had more carbon that was de-carbonized from the S45C steel matrix.

3.3. Mechanism of formation of the interface

The mechanism of the formation of three sections of the interface after during the pouring of copper a 1150°C is considered. The surface of the S45C steel rod, at appropriate temperature, was melted to form a cast welding layer; meanwhile, the heat of molten copper was transmitted to the surface of the S45C steel rod, increasing the temperature of the surface, causing de-carbonization. The cast-welding layer thus had highly concentrated carbon and a few copper atoms that had interdiffused with the iron atoms. The compound casting was placed in an isothermal furnace for two hours at 800°C to promote diffusion. At this temperature, the coefficient of diffusion of iron into the copper matrix was greater than that of copper into the iron matrix; therefore, iron atoms passed through the cast-welding layer from the matrix to the copper matrix to generate the middle layer. The width of such a reacted layer increased with time. As iron atoms diffused into the copper matrix, carbon atoms in the cast welding layer also diffused into the middle layer, forming a solid solution in the interstices of the iron unit cells; the many carbon atoms effectively prevented copper atoms from passing through the cast welding layer. Diffusion proceeded, and iron atoms in the middle layer formed $(Fe\text{-}C)_x$ with supersaturated carbon, effectively preventing copper atoms with a lower diffusion rate from entering the middle layer. Diffusion

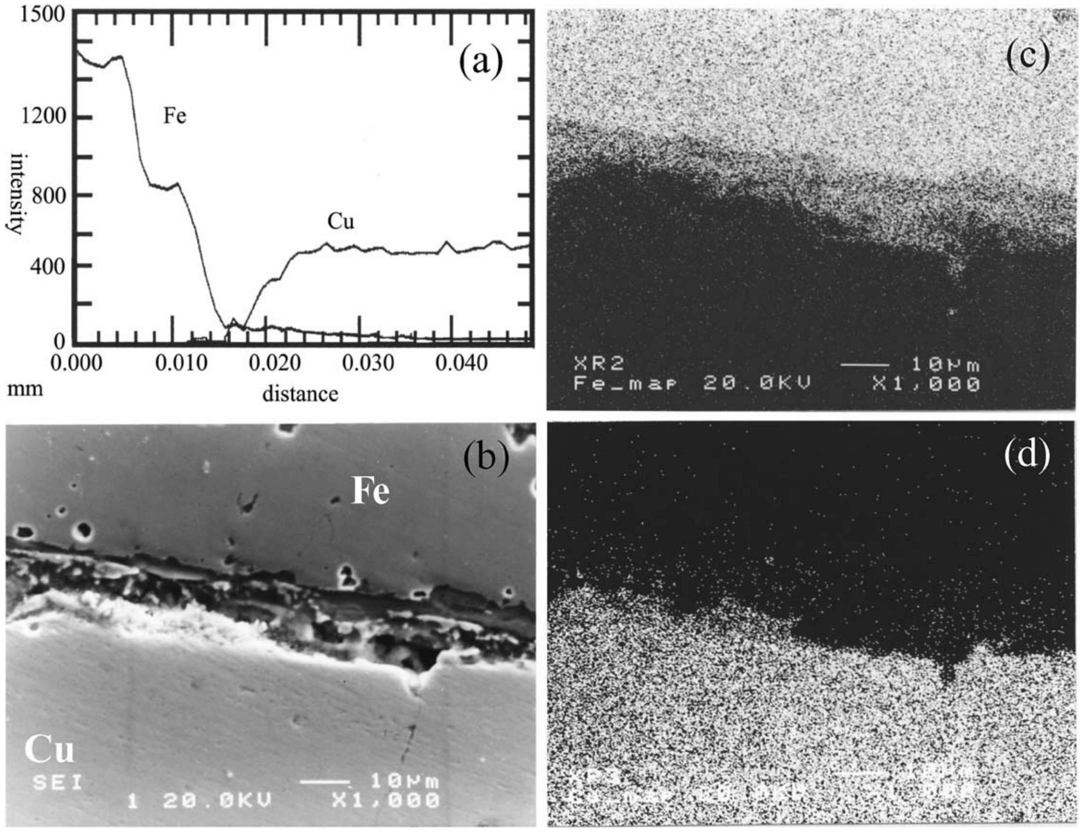

Figure 6 EPMA of S45C steel/copper compound casting: (a) line profile, (b) SEM photograph, (c) iron map, and (d) copper map.

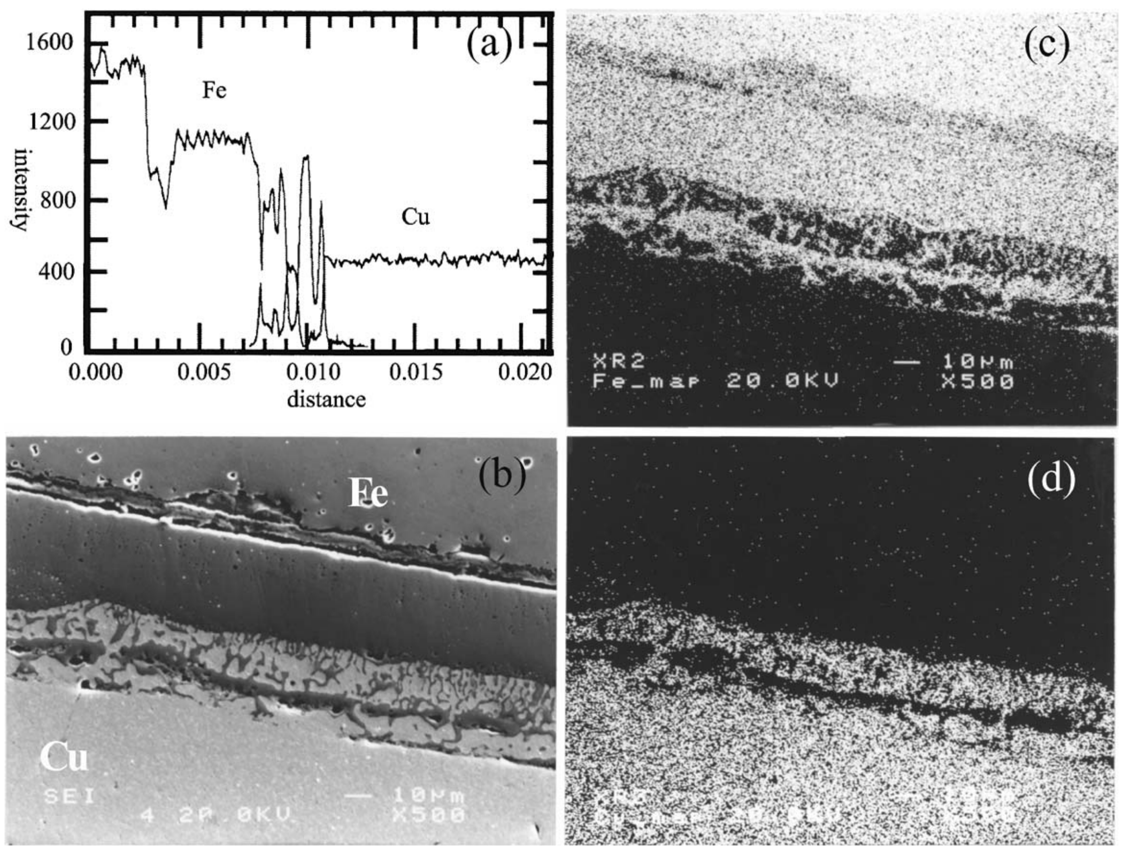

Figure 7 EPMA of S45C steel/copper compound casting after air cooling: (a) line profile, (b) SEM photograph, (c) iron map, and (d) copper map.

continued through the routes formed among the (Fe-C)$_x$ particulate in the middle layer, and iron atoms diffused into the copper matrix. Accordingly, an irregular layer was R formed. Connections and diffusing routes were established while iron atoms diffused in all directions throughout the copper matrix. Then, a secondary middle layer was also formed in the irregular layer.

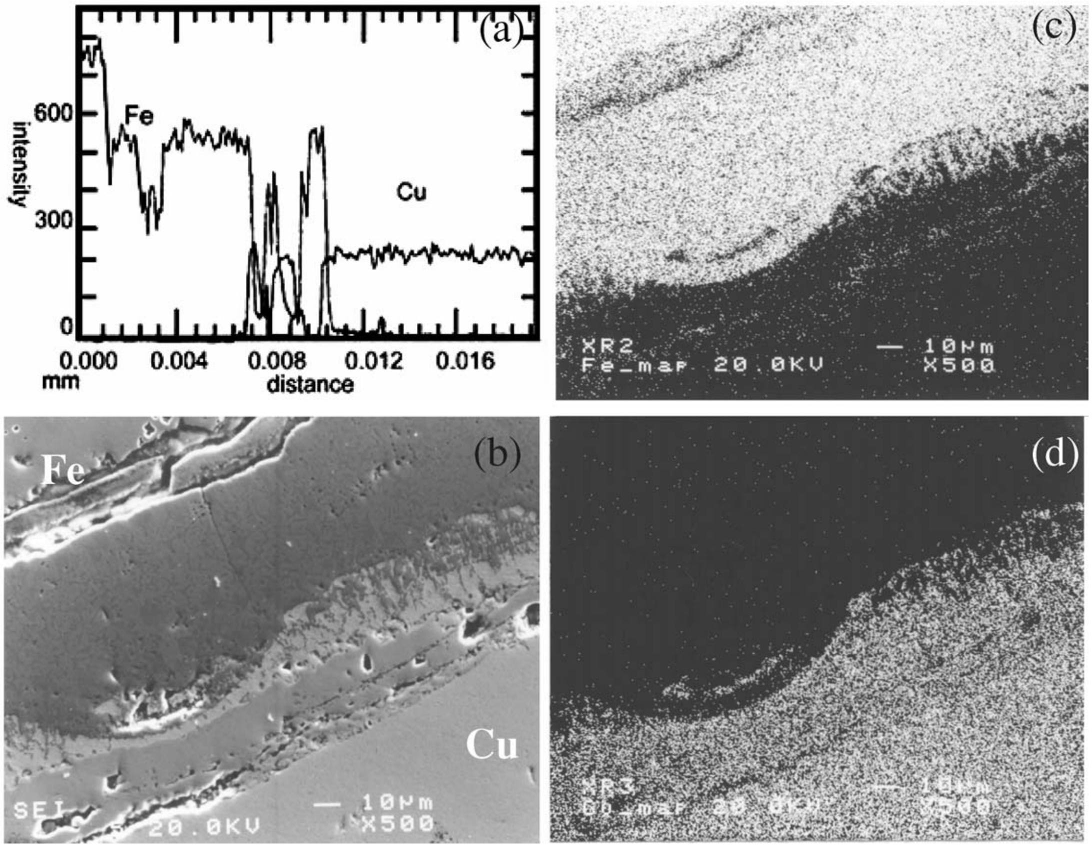

Figure 8 EPMA of S45C steel/copper compound casting after furnace cooling: (a) line profile, (b) SEM photograph, (c) iron map, and (d) copper map.

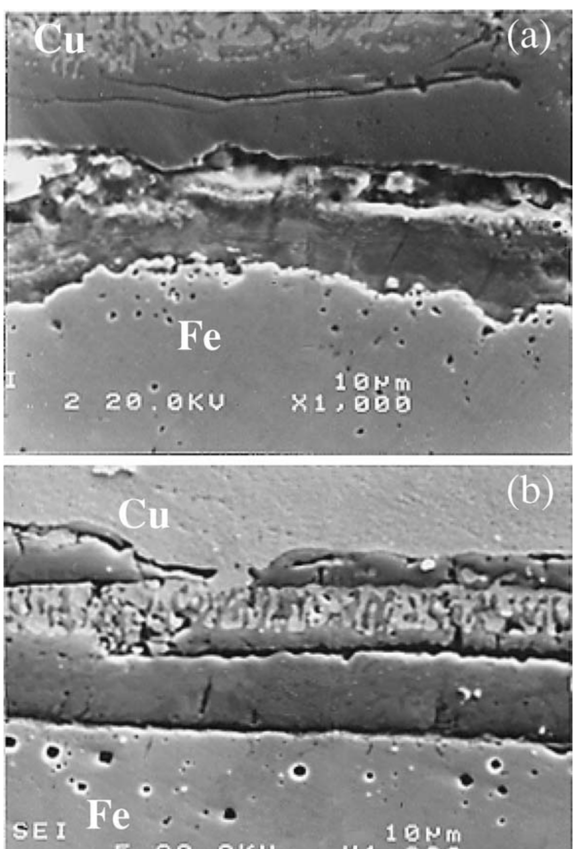

Figure 9 SEM photographs of the interface following: (a) water quenching and (b) furnace cooling.

3.4. Push-out analysis of interface

Before continuous cooling heat-treatment, the average interface shear stress of a cast-welded layer, formed using cast welding S45C steel and copper, was 6.37 Mpa. After continuous cooling heat-treatment, the average interface shear stresses of a water-quenched, oil-quenched, air-cooled and furnace-cooled cast-welding layer were 5.49, 5.68, 8.53 and 9.60 Mpa, respectively. Experimental results suggest that the interface shear stress decreases as the increasing cooling rate increases. In the current study, all four cooling processes yielded the same thermal stress $\Delta\sigma$ in the S45C steel, the magnitude of which was proportional to the temperature change ΔT, and to the change in the coefficient of thermal expansion Δc; $\Delta\sigma \sim (\Delta c)(\Delta T)$. When the thermal stress exceeds the critical value for dislocation formation, a new dislocation was introduced into the slip system in the S45C steel. Dislocations move toward the S45C-$CuFeO_2$ interface and pile up along this interface. During rapid cooling, dislocations move faster, promoting accumulation at the interface. When a shear stress, generated in the push-out test, for example, is applied to the interface, the stress at the dislocation pile-up is proportional to the number of dislocations [15]; when the interfacial shear stress reaches a threshold, the piles of dislocations coalesce into a wedge-shaped crack [16], the size of which is again proportional to the number of dislocations. Consequently, rapid cooling, such as quenching in water weakens the S45C-$CuFeO_2$ interface. Once debonding at the interface begins, it propagates in the longitudinal direction of the S45C

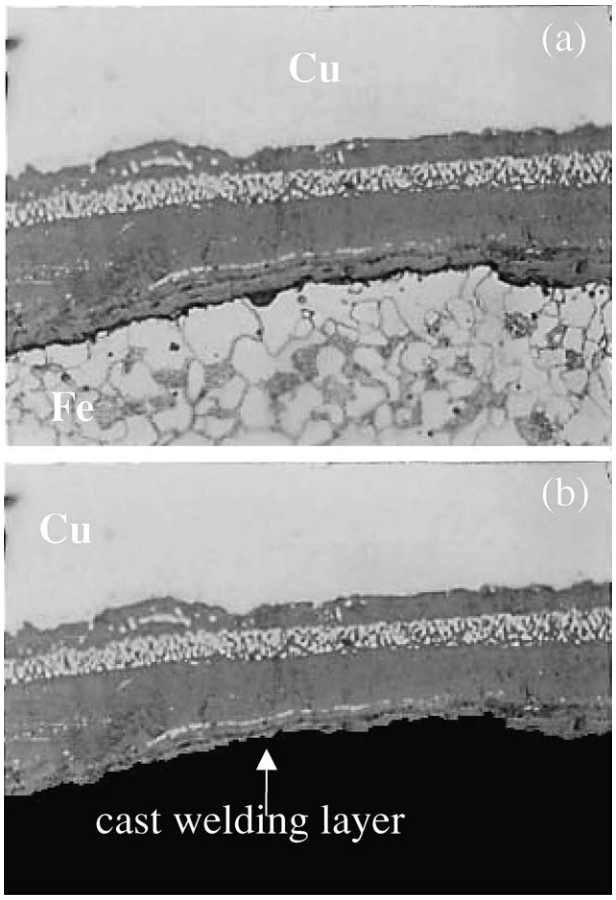

Figure 10 Push-out fracture morphology of S45C steel/copper compound casting: (a) before heated treatment and (b) after water quenching.

and frictional sliding at the interface causes subsequent motion. The Poisson effect expanded the S45C and the thermal contraction, increasing the radial compressive stress across the interface. According to Durrant *et al.* [3] the shear stress at the interface is $\tau = -\mu p$, where μ is the coefficient of friction and p is determined from radial compressive stress across at the interface. The presented experimental results imply that push-out sliding stress decreases as the cooling rate decreases because μ was the same under all heat treatment conditions; a cast-welded layer near the S45C steel matrix under any heat treatment conditions, contains a ferrite region, so the push-out sliding strength is only governed only by the radial compressive stress across the interface. The net change in temperature due to cast welding (1123°C) exceeded that due to continuous cooling heat-treatment (790°C for water quenching and 773°C for other types of quenching), so the cast welding yielded the largest the radial compressive stress across the interface, and largest corresponding sliding stress. Fig. 10 indicates that the fracturing region before heat-treatment and during heat-treating was near the steel matrix in the cast-welded layer, because this region had a high concentration of stresses.

4. Conclusions

Based on the results in this study, we conclude the following:

1. Heat treatment formed reacted layers formed in the interface. The layer near the S45C steel matrix was the cast welding layer; another close to the copper matrix was the irregular layer, and the other between these two layers was the middle layer.
2. EPMA proved that most of the iron atoms diffused into the copper matrix and only a few copper atoms diffused into the iron matrix during diffusion occurred between two matrices. X-ray diffraction showed that the chemical compounds of the interface were $CuFeO_2$ and C.
3. Furnace-cooling yielded the largest interface shear strength, and water quenching yielded the least.
4. The fractured region was near the S45C steel matrix in the cast welding layer.

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